LAYERING ASZM-TEDA CARBON BEDS OF VARYING PARTICLE SIZES TO OPTIMIZE FILTER PERFORMANCE

Richard A. Newton
Marine Team
Soldier Biological Chemical Command
Aberdeen Proving Ground, Maryland

Paulette Jones
CBR Filtration Team
Soldier Biological Chemical Command
Aberdeen Proving Ground, Maryland

Robert W. Morrison
Guild Associates Inc.
Baltimore, Maryland

ABSTRACT

This research investigated cyanogen chloride (CK) filtration performance and airflow resistance of adsorbent beds consisting of two ASZM-TEDA Carbon layers of different particle size. Adsorbent particle size affects filter performance in two ways: (1) smaller particle size results in increased chemical vapor breakthrough time and (2) larger particle size results in lower airflow resistance. CK was used as a basis for assessing filtration performance because it is a design-limiting agent for NBC filters. The results of the study showed that ASZM-TEDA Carbon beds consisting of an inlet layer of larger particle size followed by a layer of smaller particle size provided a superior trade-off of filtration performance and airflow resistance.

INTRODUCTION

Under the Joint Service General Purpose Mask (JSGPM) development program, the filter system is required to provide a high level of filtration performance and low airflow resistance, while being lightweight and small in size. The small filter size requirement is based on the vast array of military equipment to which the mask must be compatible. Much of this equipment, such as sighting devices, severely restricts the size of the filter that can be mounted on the respirator facepiece. This size restriction severely limits the options available to meet the filtration performance and airflow resistance requirements. Much of the volume of the filter is occupied by the adsorbent bed. Thus, the identification of adsorbent bed compositions that offer improved sorption agent capacity and low airflow resistance is an important aspect of JSGPM filter development. One approach to achieve improvement in filtration performance and airflow resistance is to alter the particle size of the adsorbent used in the filter. The work reported in this paper investigates the use of ASZM-TEDA Carbon in two layers of differing particle size

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to make use of the filtration performance benefit provided by small particle size while minimizing the impact of small particle size on airflow resistance.

BACKGROUND

It is well known that the protection time afforded by a filter increases as the diameter of the adsorbent particle is reduced.[1, 2] However, smaller particle size results in increased air flow resistance, thus limiting the minimum particle diameter one may use in filters for individual and collective protection application. Current military filters contain ASZM-TEDA carbon consisting of a distribution of particle sizes designated as U.S. Sieve 12x30 mesh. (The terminology 12x30 mesh refers to particles that pass through a No. 12 sieve but do not pass through a No. 30 sieve.) Military filters are filled with a homogenous blend of this carbon as a single layer. The specification for this "standard" ASZM-TEDA Carbon places requirements on the distribution of particle sizes within this 12x30 range. While this particle size provides a good balance of filtration performance with airflow resistance for many applications, 12x30 mesh may not be optimal for the very thin adsorbent beds needed for compatibility with JSGPM requirements. Thus, a 20x40 mesh particle size is being considered for the JSGPM filter to meet the requirements of high levels of filtration performance and the small filter size needed to minimize equipment interface problems. However, this reduced carbon particle size results in an undesirable increase in airflow resistance.

The positive impact of particle size on protection time and its negative effect on airflow resistance require the designer of vapor filters to search for the optimum particle size composition of the adsorbent bed. Generally, the entire adsorbent bed of a filter is comprised of a single layer of a specific particle size blend selected to obtain the best trade-off of protection and airflow resistance. However, a better trade-off of these filter performance properties should be offered through the use of a layered adsorbent bed consisting of a smaller particle size adsorbent at the outlet of the bed than that at the inlet. Since the CK reactive capacity of ASZM-TEDA Carbon tends to be essentially independent of particle size, the effect of particle size on CK filtration performance results from the size of the "mass transfer zone". (In this case "mass transfer zone" is used to indicate that portion of the adsorbent bed of a filter in which the vapor phase concentration decreases from the challenge concentration to the breakpoint concentration through the action of chemical reaction as well as mass transfer processes.) Thus, to improve filtration performance, one need only apply enough smaller particle size adsorbent to accommodate the mass transfer zone. The remainder of the bed, which is used to capacity, can be of larger particle size so as to minimize airflow resistance. The use of a smaller particle size throughout the entire carbon bed would result in a larger than necessary airflow resistance penalty.

This analysis of optimum particle size layering can become very complicated if more than two layers are considered. Obviously, the simplest layered adsorbent bed configurations consist of two layers. This paper describes the results of a study of adsorbent beds consisting of two layers of different particle size ASZM-TEDA Carbon to obtain an advantageous trade-off of filtration performance and airflow resistance. Specifically, this study maximizes protection time and minimizes airflow resistance of a 1.0-centimeter bed of carbon by varying the layers and particle sizes of ASZM-TEDA carbon. Breakthrough times and airflow resistances for non-layered 12x30 and 20x40 sized beds are provided for comparison.

Phase 1 of this study generated a matrix of CK breakthrough times and airflow resistances data for single layer beds consisting of a variety of particle sizes of ASZM-TEDA Carbon. These data were then used to formulate a test matrix for 1.0 centimeter deep layered beds consisting of two different particle sizes of ASZM-TEDA Carbon in a variety of bed depth combinations. The CK filtration performance and airflow resistance of these layered beds were measured in Phase 2 of this study. The Phase 2 data allows one to select the best combination of particle size and depth for each of two layers of ASZM-

TEDA Carbon to obtain an optimal trade-off of filtration performance and airflow resistance for the JSGPM primary filter.

EXPERIMENTAL APPROACH

In Phase 1, ASZM-TEDA Carbon in seven particle size ranges and five bed depths was tested for CK filtration performance and airflow resistance. CK filtration was measured at an airflow velocity of 6 cm/sec, which is equivalent to 50 liters per minute through a JSGPM filter concept. Airflow resistance was measured at this same airflow velocity of 6 cm/sec. To obtain the narrow particle size ranges, 12x30 mesh size ASZM-TEDA Carbon from Calgon Carbon Corporation, Pittsburgh, Pennsylvania (Lot N99916CK) was ground and sieved. The broader particle size ranges were then prepared by combining appropriate narrow particle size ranges in equal mass proportions. This approach resulted in a more uniform particle size distribution within each range than that obtained by direct sieving of the ground carbon.

The ASZM-TEDA Carbon was tested in 4.1 cm inside diameter glass tubes with baffled support screens. As the carbon depths are thin, great care was taken to fill uniform beds in the tubes. The carbon was loaded in the tubes using the "snowstorm" filling technique. After loading, the carbon bed was leveled using the 4 cm diameter plastic cylinder. Care was taken such that the tubes were neither jolted nor tilted. The beds were checked diligently to ensure that each was uniform in thickness. Small differences in the uniformity of the bed depth could obscure the benefits to airflow resistance and breakthrough time gained via layering of the carbon bed. If any shifting of the carbon granules occurred, the carbon sample was reloaded into the tube.

In the Phase 2 layered bed evaluations, the inlet layer was 12x30 mesh ASZM-TEDA Carbon. The outlet layer was one of five different smaller particle sized ASZM-TEDA Carbons. The total bed depth was fixed at 1.0 cm with three variations of bed depth combinations for the component layers. The outlet layer (bottom layer in tube) of carbon was loaded and leveled first, at the specified bed depth using aforementioned filling techniques. The inlet carbon layer (top layer in tube) was loaded and leveled on top of the outlet layer using the same procedures. Great care was again taken to maintain bed packing uniformity of the outlet layer while loading and leveling the inlet layer.

TEST PARAMETERS

Phase 1 - Single Particle Sized Beds:

CK Filtration Performance

CK Challenge Concentration:	4,000 mg/m^3
CK Effluent Concentration:	8 mg/m^3
Relative Humidity:	80% (preconditioned to 80%)
Temperature:	25^{o}C
Airflow Velocity:	6 cm/sec
Bed Depths and Carbon Particle Sizes:	

U.S. Sieve Particle Size of Carbon	Average Particle Diameter (millimeters)	Bed Depths (centimeters)				
		0.4	0.6	0.8	1.0	1.2
12x20	1.26		X	X	X	X
12x30	1.02		X	X	X	X
20x30	0.718		X	X	X	X
20x40	0.631	X	X	X	X	
30x40	0.508	X	X	X	X	
30x50	0.446	X	X	X	X	
40x60	0.335	X	X	X	X	

X = Test Performed

Phase 2 - Multiple Particle Sized Beds:

CK Filtration Performance

CK Challenge Concentration: 4000 mg/m^3
CK Effluent Concentration: 8 mg/m^3
Relative Humidity: 80% (preconditioned to 80%)
Temperature: 25°C
Airflow Velocity: 6 cm/sec
Particle Size (Inlet layer): 12x30 mesh ASZM-TEDA (Lot N99916CK)
Particle Sizes (Outlet layer): 20x30, 20x40, 30x40, 30x50, 40x60 ASZM-TEDA
Bed Depths: 1.0 cm total (Inlet layer of 12x30 ASZM-TEDA followed by one of the smaller grain sizes per table)

		Outlet (Bottom) Layer Thickness		
		0.2 cm	0.4 cm	0.6 cm
Inlet (Top) Layer Thickness	0.4 cm			X
	0.6 cm		X	
	0.8 cm	X		

X = Test Performed

Airflow Resistance

Temperature: 25°C
Airflow Velocity: 6.0 cm/sec
Bed Depths and Particle Sizes: As indicated in table above

RESULTS AND DISCUSSION

Single Particle Size Beds

Figure 1 shows the life thickness curves for the data generated for the single particle-size adsorbent beds. Each data point represents the average of duplicate measurements. The curves show a very strong relationship between particle size and CK breakthrough time at an effluent concentration of 8 mg/m^3 for all bed depths. As expected, for a given bed depth, the smallest average particle size carbon has the longest breakthrough time. This relationship exists because of the greater external surface of smaller particle size adsorbent results in greater mass transfer of CK from the airflow passing through the bed to the pore structure containing the reactive impregnants on the carbon.

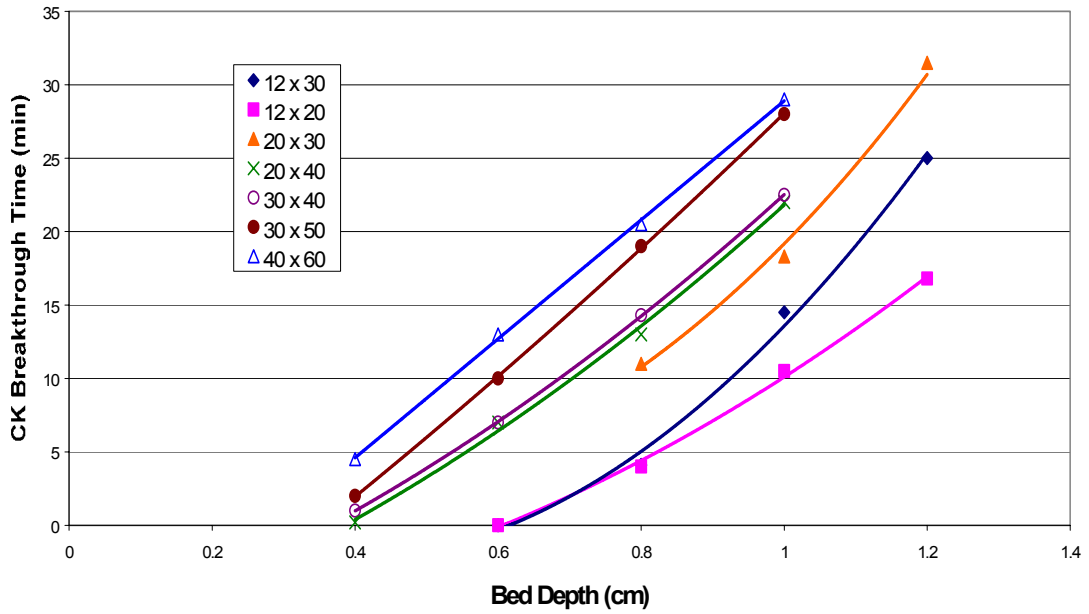

Figure 1. ASZM-TEDA Life Thickness Curves.
(Airflow Velocity = 6 cm/sec; RH = 80/80; Temperature = 25°C)

Table 1 contains the measured airflow resistances in millimeters of water for 1.0 and 2.0-centimeter carbon beds at 6.0 centimeters per second velocity for the various mesh size carbons. As expected the smaller particle sized carbons had greater airflow resistance. This trend results from increased viscous drag exerted on the airflow by the greater external surface area that exists in a bed of smaller particles.

Table 1. Airflow Resistances of Various Mesh Sizes of ASZM-TEDA Carbon

Particle Size	Airflow Resistance (mm H_2O)	
	1.0 cm bed depth	2.0 cm bed depth
12x30	3.43	6.48
12x20	2.79	5.33
20x30	5.46	10.03
20x40	7.87	14.73
30x40	9.02	16.38
30x50	13.59	25.65
40x60	17.65	35.94

Layered Particle Size Beds

The airflow resistances for the layered beds were calculated by summing the values obtained by multiplying layer depth by the appropriate airflow resistance for a 1.0 cm. deep bed from Table 1. For example, a bed with 0.4 cm. of 12x30 mesh carbon and 0.6 cm. of 30x40 carbon has a calculated airflow resistance of 6.78 millimeters water (0.4 cm x 3.43 mm water per cm of bed + 0.6 cm x 9.02 mm water per cm of bed). The results of these airflow resistance calculations are provided in the middle column of Table 2. The right hand column provides the measured CK breakthrough times for the carbon beds.

Table 2. Calculated Airflow Resistances and Breakthrough Times for the Layered Beds

Carbon Sieve Size and Bed Depth Fractions (cm)	Airflow Resistance (millimeters H2O)	Breakthrough Time (minutes)
0.4 cm (12x30) 0.6 cm (40x60)	11.96	27.5
0.4 cm (12x30) 0.6 cm (30x50)	9.52	27.0
0.6 cm (12x30) 0.4 cm (40x60)	9.12	26.5
1.0 cm (20x40) JSGPM Filter	7.87	22.0
0.6 cm (12x30) 0.4 cm (30x50)	7.50	23.0
0.4 cm (12x30) 0.6 cm (30x40)	6.78	27.5
0.8 cm (12x30) 0.2 cm (40x60)	6.27	21.5
0.4 cm (12x30) 0.6 cm (20x40)	6.09	22.0
0.6 cm (12x30) 0.4 cm (30x40)	5.66	22.5
0.8 cm (12x30) 0.2 cm (30x50)	5.46	22.0
0.6 cm (12x30) 0.4 cm (20x40)	5.21	18.0
0.4 cm (12x30) 0.6 cm (20x30)	4.64	18.5
0.8 cm (12x30) 0.2 cm (30x40)	4.54	17.5
0.8 cm (12x30) 0.2 cm (20x40)	4.31	18.0
0.6 cm (12x30) 0.4 cm (20x30)	4.24	18.5
0.8 cm (12x30) 0.2 cm (20x30)	3.83	16.0

The data show that by composing adsorbent beds of layers of large particles at the inlet and small particles at the outlet, it is possible to increase CK breakthrough time as well as lower airflow resistance relative to that exhibited by the same bed depth of all 20x40 particles. Table 2 shows six different bed configurations where the airflow resistance is lower than that of an all 20x40 particle size bed and the breakthrough time is at least equal to the 22 minutes of the 20x40 particle size bed. An especially noteworthy combination of CK breakthrough performance and airflow resistance was obtained by layering 0.4-centimeter of 12x30 particles at the bed inlet and 0.6-centimeter of 30x40 particles at the bed outlet. The aforementioned-layered bed provides an airflow resistance of 6.78 millimeters of water compared to 7.87 mm water for a homogeneous bed of 20x40 particles. Additionally, layering of the carbon bed increases the time at which the effluent concentration reaches 8 mg/m^3 from 22 minutes, for a homogeneous bed of 20x40 particles, to 27 minutes for the layered bed.

The basic parameters that dictate the filtration performance of ASZM-TEDA carbon against CK are the reactive capacity of the adsorbent and the rate that adsorbent removes the CK from the airflow passing through the adsorbent bed. The capacity is dictated in this case by the amount of reactant on the carbon. Since each of the different particle-sized carbons were obtained by grinding and sieving operation from the same parent 12x30 mesh ASZM-TEDA Carbon, the reactive capacity of the impregnants on all the different particle size carbons is the same. Thus, the observed differences in CK breakthrough time do not result from differences in reactive capacity toward CK.

The differences observed in breakthrough time results from differences in CK mass transfer from the carbon bed airflow to the impregnants in the pore structure of the carbons. This increased mass transfer rate is a combination of increased mass transfer external to the carbon particle as well as increased mass transfer internal to the carbon granules as the CK diffuses to the sites of the impregnants within the pore structure of the carbon. For a given adsorbent bed volume, smaller sized particles have a larger external area than do larger sized particles. Thus, with more external area in the smaller particle size bed through which to diffuse CK through the boundary layer of relatively stagnant air that exists around each particle, increased CK removal from the airflow occurs. Once the CK molecules have diffused to the pore structure of the carbon, their access to the reactive impregnants is greater for smaller particle sized

carbon. The internal distances within smaller particle sizes are such that the distance that a molecule of CK must diffuse within the pore structure is less. Thus, with the faster access to the impregnants, an increased rate of CK destruction by the impregnants results. The extent that internal mass transfer contributes to the removal of CK by a reactive adsorbent has been addressed by Friday and Mahle[3].

The filtration performance benefit resulting from the use of layered beds of different particle size is only obtained by placing the smaller particle size adsorbent at the exit of the bed. In so doing, as the sorption wave passes through the carbon bed, the wave sharpens in the small particle size portion of the bed. This results in a shorter "mass transfer zone" and leaves more adsorbent in the "capacity zone" of the bed to be used to full capacity. Since the capacity of the carbon is independent of particle size, the use of larger particle size adsorbent in this capacity zone at the inlet of the bed does not adversely impact on filtration performance, but does provide the benefit of lower airflow resistance.

CONCLUSIONS

Adsorbent beds consisting of layers of different particle size offer a superior trade-off of CK filtration performance versus airflow resistance as compared to a bed of a single particle size.

The exit layer of smaller particle size adsorbent should be of sufficient depth to include essentially the entire mass transfer zone, thus maximizing the filtration capacity of the adsorbent bed.

A bed consisting of a 0.4 centimeter layer of 12x30 mesh carbon followed by a 0.6 centimeter layer of 30x40 mesh carbon offered a 25% increase in CK breakthrough time and a 14% reduction in airflow resistance as compared to those characteristics exhibited by a 1.0 centimeter deep bed of all 20x40 mesh ASZM-TEDA Carbon.

REFERENCES

1. Newton, Richard A. and David K. Friday, "Breakthrough Times of Cyanogen Chloride on Layered and Mixed Beds of ASC Whetlerite", Proceedings of the 1990 Chemical Research Development and Engineering Center Scientific Conference on Chemical Defense Research, CRDEC Technical Report AD-B-165-717, pp 547-551.

2. Keller, George E., et al., "Adsorption", in Handbook of Separation Process Technology, edited by Ronald W. Rousseau, John Wiley & Sons (1987).

3. Friday, David K. and John J. Mahle, "Analysis of a Fixed Bed Adsorptive Reactor Using Cyanogen Chloride ASC Carbon System", CRDEC Technical Report TR-022, January 1989.